



# **Comparative Stress Corrosion Cracking and General Corrosion Resistance of Annealed and Hardened 440C Stainless Steel—New Techniques in Stress Corrosion Testing**

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## TECHNICAL PUBLICATION

# COMPARATIVE STRESS CORROSION CRACKING AND GENERAL CORROSION RESISTANCE OF ANNEALED AND HARDENED 440C STAINLESS STEEL—NEW TECHNIQUES IN STRESS CORROSION TESTING

## 1. INTRODUCTION

Current methods for evaluating the susceptibility of a material to stress corrosion cracking (SCC) involve loading specimens in tension to varying levels of the material's yield strength, placing the specimens in a corrosive environment, and monitoring the time to failure. Specimen configurations range from smooth bar tensile, to C-ring, to 3-point bend. At the Marshall Space Flight Center, the constant strain, smooth bar tensile specimen is used almost exclusively. Specimens are typically loaded in quintuplicate to 0, 25, 50, 75 and 90 percent of the material's yield strength and tested in three environments: High humidity (100-percent relative humidity (R.H.), 95 °F), 5-percent NaCl salt fog and 3.5-percent NaCl alternate immersion. Evaluation of material susceptibility to SCC is based on time to failure at each stress level.

While these techniques for SCC testing have worked very well over the years, there are some shortcomings. First, these methods yield only qualitative data. A true threshold stress for SCC is not obtained because this threshold, typically designated  $K_{ISCC}$ , requires consideration of both the bulk stress in the specimen and the concentration of this stress around defects in the specimen. Crack growth in the smooth bar specimens occurs when a corrosion pit or other defect grows to a critical size, which decreases with increasing applied stress. The size of this defect is not readily quantified using smooth bar specimens. The time to failure at a particular stress level becomes more a question of defect initiation and growth to critical size rather than initiation of crack growth. In the study of SCC, we are, from a design standpoint, much more concerned with that combination of flaw size, stress and environment which will cause an existing defect to grow than with the time required to initiate a flaw. Second, these techniques are long duration, requiring at least 30 days and sometimes up to 6 months to generate useful data. Finally, these techniques tend to be very labor intensive and costly (five specimens at five stress levels in three environments yields 75 specimens).

Alternative techniques which involve the use of fatigue-precracked single-edge notched bend (SE(B)) specimens, yield quantitative data. Specimen defect size is readily determined from compliance equations. Examples include the bolt-loaded wedge-opening-loading (WOL) and the cantilever beam techniques. While it is possible with these techniques to obtain a true threshold for SCC ( $K_{ISCC}$ ), the test duration is prohibitively long, taking up to a year or more. In addition, there is some question of whether a true threshold is actually obtained, particularly in the WOL technique, since self loading of the crack due to corrosion product accumulation can yield conservatively low results.

Two new techniques for evaluating susceptibility to SCC are being investigated at MSFC under the Center Director's Discretionary Fund Program. Both techniques involve the use SE(B) specimens, similar to those used in fracture toughness testing. Specimen loading for both techniques requires controlled loading on a universal test machine. The first method involves loading fatigue-precracked specimens in a step-wise fashion exposed to a corrosive environment. Each step increase in load corresponds to an increase in crack-opening-displacement (COD), monitored with a clip gauge. At low stress intensity, the COD versus time curve will have a zero slope at each step. The stress intensity corresponding to subcritical crack growth, calculated from the applied load and the initial flaw size, is indicated by a nonzero slope. In other words, the COD is increasing while the applied load remains unchanged, with crack growth resulting in the increase in COD. The slope of this curve typically increases with each step until fracture occurs, indicating a larger crack growth rate with each increase in load.

The other method which utilizes precracked SE(B) specimens involves loading the specimens at constant load rate in a corrosive environment. A plot of load versus COD, similar to but slower than an ASTM E-399 fracture toughness test, yields a straight line over most of the range tested. Crack growth is indicated by deviations in this curve from linearity at higher stress intensity levels. Since these deviations occur at stress intensity levels well below  $K_{IC}$  (the stress intensity required for unstable crack growth or fracture in air), it is assumed that their cause is sub-critical crack growth due to the corrosive environment; stress corrosion cracking. Deviations from linearity are calculated for 1-, 2-, and 4-percent offset from the straight line. The stress intensity corresponding to the load at which these deviations occur is taken as the stress intensity required for crack growth. Smaller offsets give more conservative results.

It is fortunate that at the time work on the CDDF began, a critical SCC-related issue on the *International Space Station* program was being evaluated. This issue presented an opportunity for these new techniques to be tested, and compared to the conventional techniques used at MSFC. A brief summary of the problem is described below.

The current design for outer and inner bearing clamps and sleeves on the SARJ Utility Transfer Assembly (UTA) specifies the use of 440C corrosion-resistant steel. This material was selected primarily because its coefficient of thermal expansion produces a favorable match in the press fit design. Since the high hardness normally associated with 440C is not required, the material is being used in the annealed condition. Hardened 440C exhibits very poor resistance to SCC. Data for the annealed material are not available. There is legitimate concern that the annealed material may be even more susceptible to SCC than the hardened material, and approval of annealed 440C for this application will require demonstration of this material's resistance to SCC. All parties agreed that an SCC evaluation of the two materials should be conducted. If the results demonstrate that the annealed material exhibits SCC resistance equal to or better than that of the hardened material, the use of the annealed material on the UTA will be accepted. Part of this evaluation also involves the general corrosion resistance of the two materials.

## **2. OBJECTIVE**

To test and compare the SCC resistance and general corrosion characteristics of 440C stainless steel in the annealed condition to that in the hardened and tempered condition.

### 3. PROCEDURES

Five types of testing were performed:

- (1) General corrosion testing in 100-percent humidity,
- (2) General corrosion testing using the direct current (DC) electrochemical method of polarization resistance,
- (3) Constant strain, stress corrosion testing in 100-percent humidity,
- (4) Stress corrosion testing using the incremental step loading technique, and
- (5) Stress corrosion testing using the constant strain rate method.

#### 3.1 General Corrosion Testing

Two each, hardened and annealed panels of 440C, 3 in.×4 in. ×3/16 in., were machined on one face. All four samples were then degreased in hexane. One each, hardened and annealed panel, was then grease-plated by dipping into a 50:1 solution of PF 5070 solvent and Braycote 601 grease. After allowing 2 hours for the solvent to flash, all four panels were placed in 100-percent humidity at 95 °F, at an angle of approximately 15° from vertical for 30 days. The panels were then removed and photographed.

#### 3.2 Electrochemical Corrosion Testing

Electrochemical corrosion rate measurements were made in a 3.5-percent NaCl solution using the direct current method of polarization resistance (PR). Flat plates of 440C used in the general corrosion testing were remachined to provide a fresh surface for electrochemical testing. An EG&G-PARC model 342 potentiostat, along with EG&G-PARC model 352 software, was used to collect and analyze the PR data. The potentiostat instrumentation automatically corrected the data for IR drop during the scan. The potential applied varied from -20 to +20mV on either side of the corrosion potential,  $E_{CORR}$ , and the data points (current and potential) were recorded in 1/4-mV increments. The PR data were analyzed using the program POLCURR<sup>1</sup>. The theory for the PR technique has been described previously<sup>2</sup>. Data were taken every other day for a period of 2 weeks.

#### 3.3 Constant Strain Stress Corrosion Testing

Nine each, hardened and annealed, smooth bar tensile specimens (fig. 1) of 440C were provided for SCC testing. Specimens were machined from round bar stock in the transverse orientation. Three of each were tensile tested in air to obtain mechanical properties. The remaining samples were degreased in hexane and loaded in precipitation hardened steel loading frames. Three each of the six remaining samples were stressed to 30 ksi. The remaining samples were stressed to 43.6 ksi. The loading frame was isolated from the samples using mascoat no. 2 by Western Coating Company. The higher stress level represents 90 percent of the yield strength of the annealed material. This high stress level was chosen in the hopes that failures in one or both of the materials would be realized in a reasonable amount of time. For comparative purposes, the same stress was applied to both the annealed and hardened materials, even though the strength of the hardened material is greater. All twelve stressed specimens were then placed in 100-percent humidity at 95 °F.



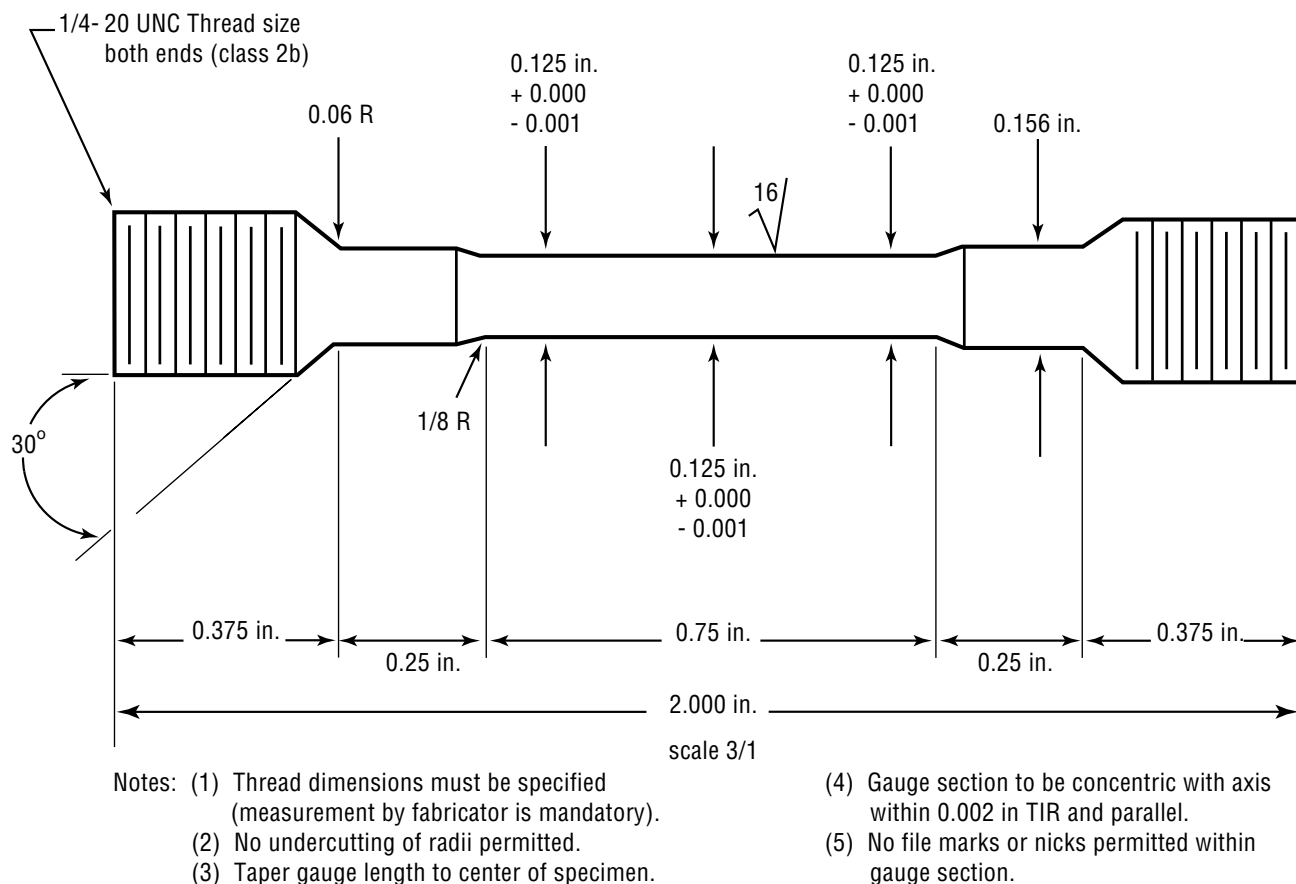


Figure 1. Round tensile specimen for constant strain stress corrosion testing.

### 3.4 Stress Corrosion Cracking Using Precracked SE(B) Specimens

Precracked specimens were machined from round bar stock, such that the C-R orientation (crack plane perpendicular to bar circumference, crack growth in radial direction) per ASTM E-399-90 was obtained. Eight each, hardened and annealed SE(B) specimens (fig. 2) were degreased in hexane in preparation for testing in a 22,000-lb. MTS servohydraulic test machine. One each of these samples was used to measure the fracture toughness of the two materials. The remaining samples were used for SCC testing by the incremental loading technique and the constant strain rate technique. Some of the SE(B) specimens were accidentally broken during the precracking procedure. This is due to the extremely delicate nature of precracking such a hard material as 440C.



crack opening displacement. All but one test was performed with 4-hour hold times and 2-ksi-in.<sup>1/2</sup> steps. The remaining hardened sample (specimen H1) was tested with 4-hour hold times and 0.5-ksi-in.<sup>1/2</sup> steps. In each test, loading started at zero. The load required to produce a desired stress intensity was based on the measured fatigue crack length and calculated using the following equation, which is specific to rectangular 3-point bend specimens:

$$K_Q = \frac{P_Q \cdot S \cdot f(a/W)}{B \cdot W^{3/2}} \quad (1)$$

where

$$f(a/W) = \frac{\sqrt{3(a/W)} \cdot [1.99 - (a/W)(1 - a/W)(2.15 - 3.93(a/W) + 2.7(a/W)^2)]}{2(1 + 2(a/W)(1 - a/W))^{3/2}} \quad (2)$$

and

$K_Q$	=	critical stress intensity (ksi-in. <sup>1/2</sup> )
$P_Q$	=	applied load (kip)
$B$	=	specimen thickness (in.)
$S$	=	span (in.)
$W$	=	specimen width (in.)
$a$	=	crack length (in.)

all dimensions per ASTM E-399-90.

The actual stress intensity was corrected after the test using average fatigue crack length measurements from the fractured sample per ASTM E 399-90. All tests were run to failure.

### 3.4.3 The Constant Load Rate Technique

One sample each of the annealed and hardened material was tested in 3.5-percent NaCl solution at pH= 6.5-7.2 using this method. The instrumentation used was the same as that used for the incremental loading technique. The rate of increase in stress intensity for both tests was 0.05 ksi-in.<sup>1/2</sup>/minute. The 1-, 2-, and 4-percent offsets were calculated by first defining a linear portion of the curve as the base slope, usually representing over 50 percent of the middle portion where the curve is most linear. A minimum correlation coefficient of 0.995 was taken as reasonable linearity for this section of the curve. Next, the slope and average load were calculated for 10-percent increments of the entire load versus COD curve, with each increment overlapping the previous and next increments by 5 percent (e.g., 0-10 percent, 5-15 percent, 10-20 percent ...). The offset for each increment was calculated by comparing the slope of the increment to the slope of the base. The average load for each increment was also calculated. These values were tabulated and the load corresponding to 1-, 2-, and 4-percent offset was calculated by interpolation. These loads were then converted to stress intensities using equations 1 and 2 and the fatigue crack lengths measured after specimen failure.

After testing, specimens were cleaned in a 50/50 solution of lactic and phosphoric acids until corrosion products were removed. The specimens were then examined and photographed in a scanning electron microscope.

## 4.0 RESULTS

### 4.1 General Corrosion Testing

Results from 100-percent humidity exposure of bare and grease-plated panels are shown in figure 3. It is noted that on the fourth day of exposure, the bare, annealed panel was found in the horizontal position in the test chamber. Since this day was a Monday, it is possible that the sample was in this position for 3 days. The severity of corrosion noted on this sample compared to the hardened sample may be due to this event. Grease-plated samples of the annealed and hardened material (fig. 4) show virtually no difference in corrosion resistance.

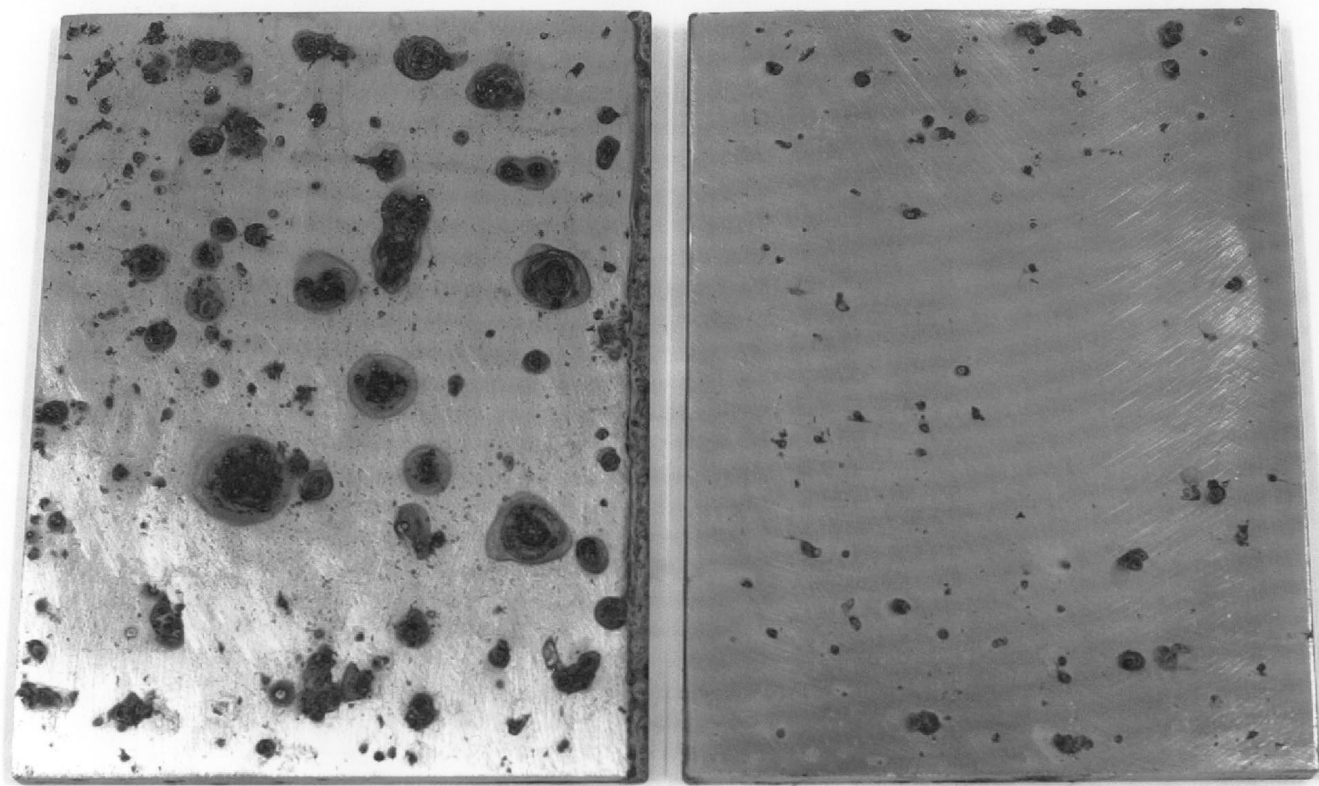


Figure 3. Annealed and hardened 440C stainless steel after 30 days exposure to 100-percent R.H. at 95 °F.

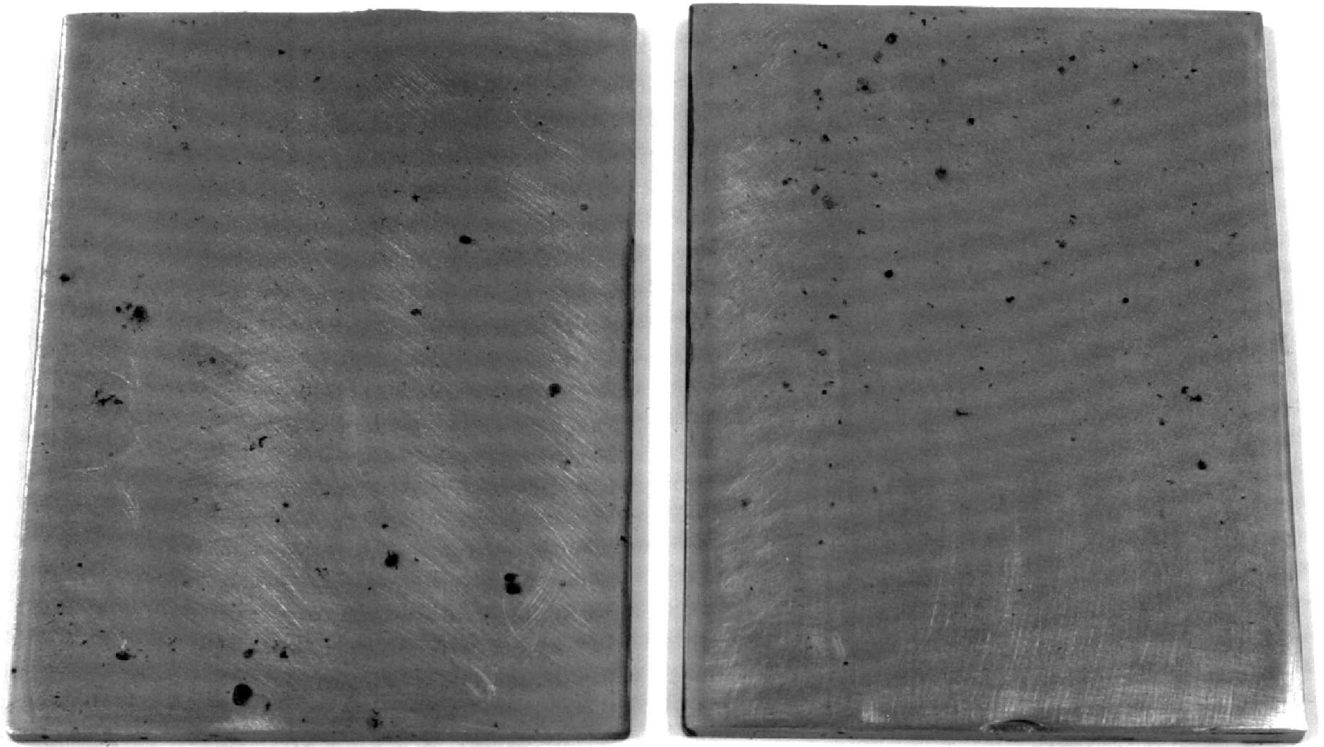


Figure 4. Annealed and braycote 601 grease plated, and hardened and braycote 601 grease plated 440C stainless steel after 30 days exposure to 100-percent R.H. at 95 °F.

## 4.2 Electrochemical Corrosion Testing

Results for the electrochemical testing were taken directly from the POLCURR output, which gives the calculated corrosion current density, in  $\mu\text{Amp}/\text{cm}^2$ , for a particular experiment. These values were converted, using the equivalent weight of 440C, into corrosion rates in mils per year (mpy). Corrosion rates for both materials are plotted as a function of time in figure 5. The average corrosion rates during the 2 weeks of testing were 3.01 mpy and 0.325 mpy for the annealed and hardened materials, respectively. In addition, the slopes of the curves are 0.0550 mpy/day and  $-0.0132$  mpy/day for the annealed and hardened materials, respectively, indicating generally increasing and decreasing trends for the corrosion rate. An example of an individual polarization resistance run is given in the appendix.

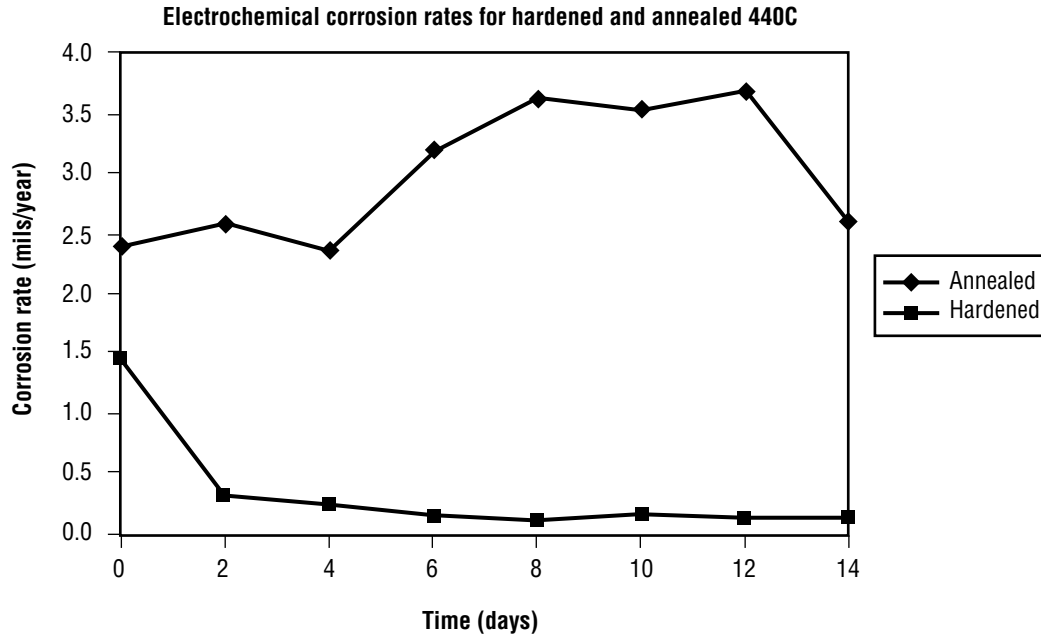


Figure 5. Electrochemical corrosion rates for hardened and annealed 440C.

### 4.3 Constant Strain Stress Corrosion Testing

The original test plan required testing in 100-percent humidity at 95 °F for 60 days. At the end of 60 days only one failure was noted: a hardened sample at 43.6 ksi failed after 1 day. These results were considered inconclusive for the purpose of determining relative SCC resistance of the two heat treatments. The test was therefore extended to 6 months. Results are given in table 1.

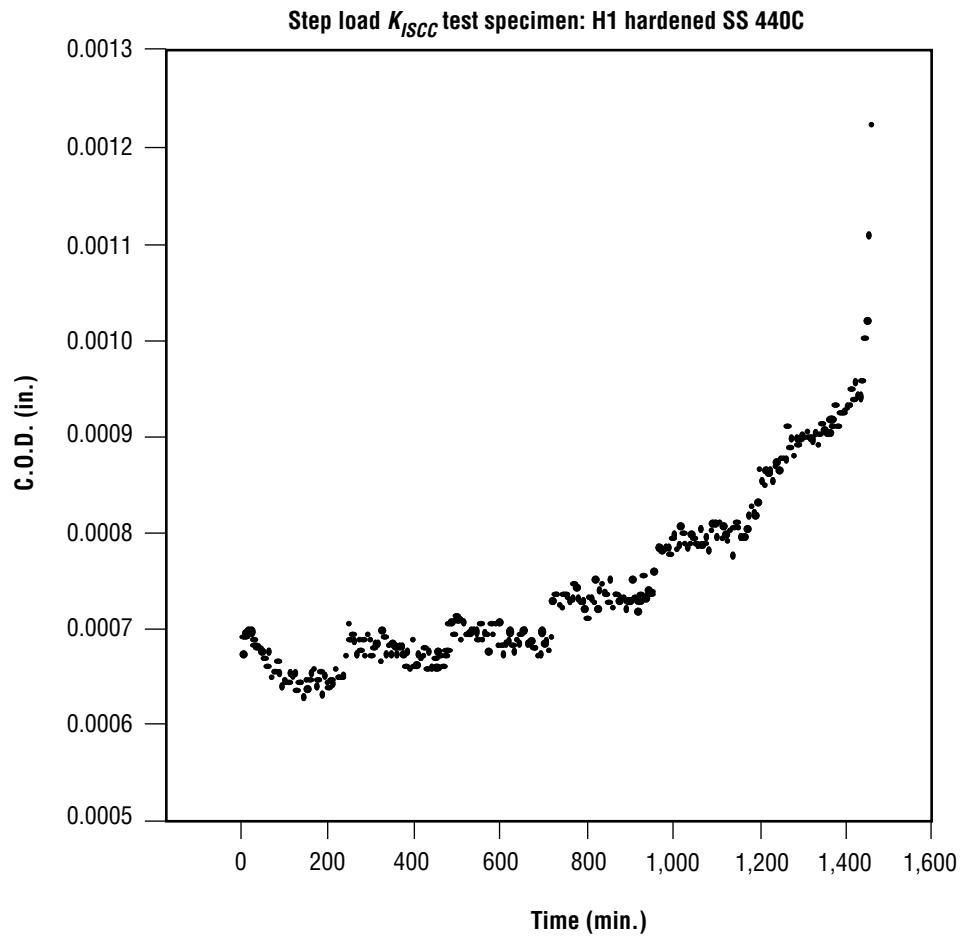
### 4.4 Stress Corrosion Cracking Using precracked SE(B) Specimens

#### 4.4.1 Fracture Toughness Testing

Results from fracture toughness testing indicate  $K_{IC}$  values of 19.4 and 25.1 ksi-in.<sup>1/2</sup> for the hardened and annealed materials, respectively. The load versus COD plots corresponding to these values are given in the appendix.

#### 4.4.2 The Incremental Loading Technique

Data generated for the hardened material using the incremental step load method are shown in figures 6-8. In figure 6, the very small step size used for specimen H1 demonstrated some problems with noise and drift in the COD gauge. This problem was not entirely resolved, but was improved using the 2 ksi-in.<sup>1/2</sup> step size for specimens H2 and H7 in figures 7 and 8, respectively. Step load data for the annealed material is shown in figures 9 and 10. Again, some drift in COD response is noted. However, in all five plots, the onset of crack growth is easily discerned within one or two steps, making the corresponding stress intensity discernible to within 2 ksi-in.<sup>1/2</sup>.



<b>Step</b>	<b>Stress intensity (ksi<math>\sqrt{in}</math>)</b>
1	5.01
2	5.51
3	6.00
4	6.50
5	7.00 (Cracking starts)
6	7.50
7	8.01 (Specimen broke)

Figure 6. Incremental step load response of specimen H1 in 3.5-percent NaCl.

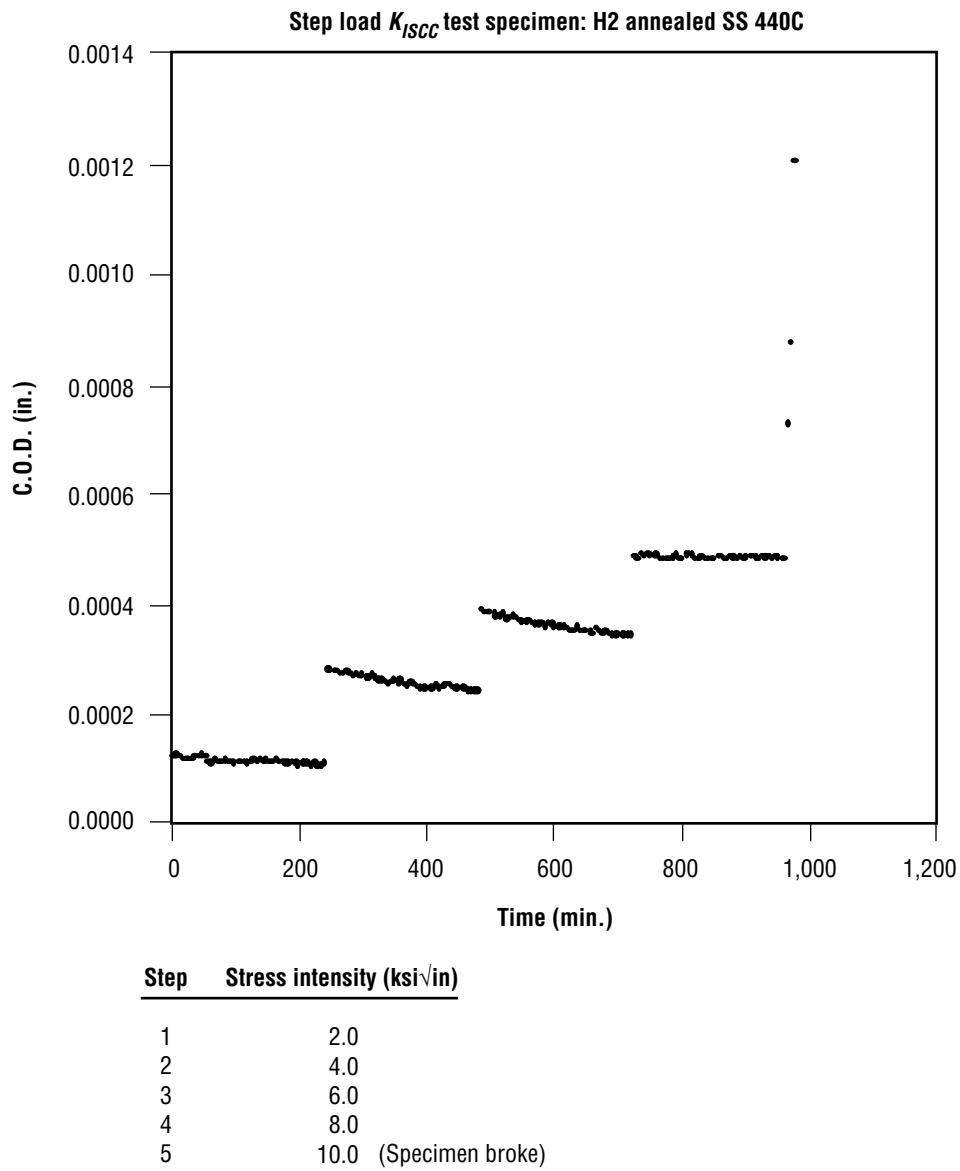


Figure 7. Incremental step load response of specimen H2 in 3.5-percent NaCl.



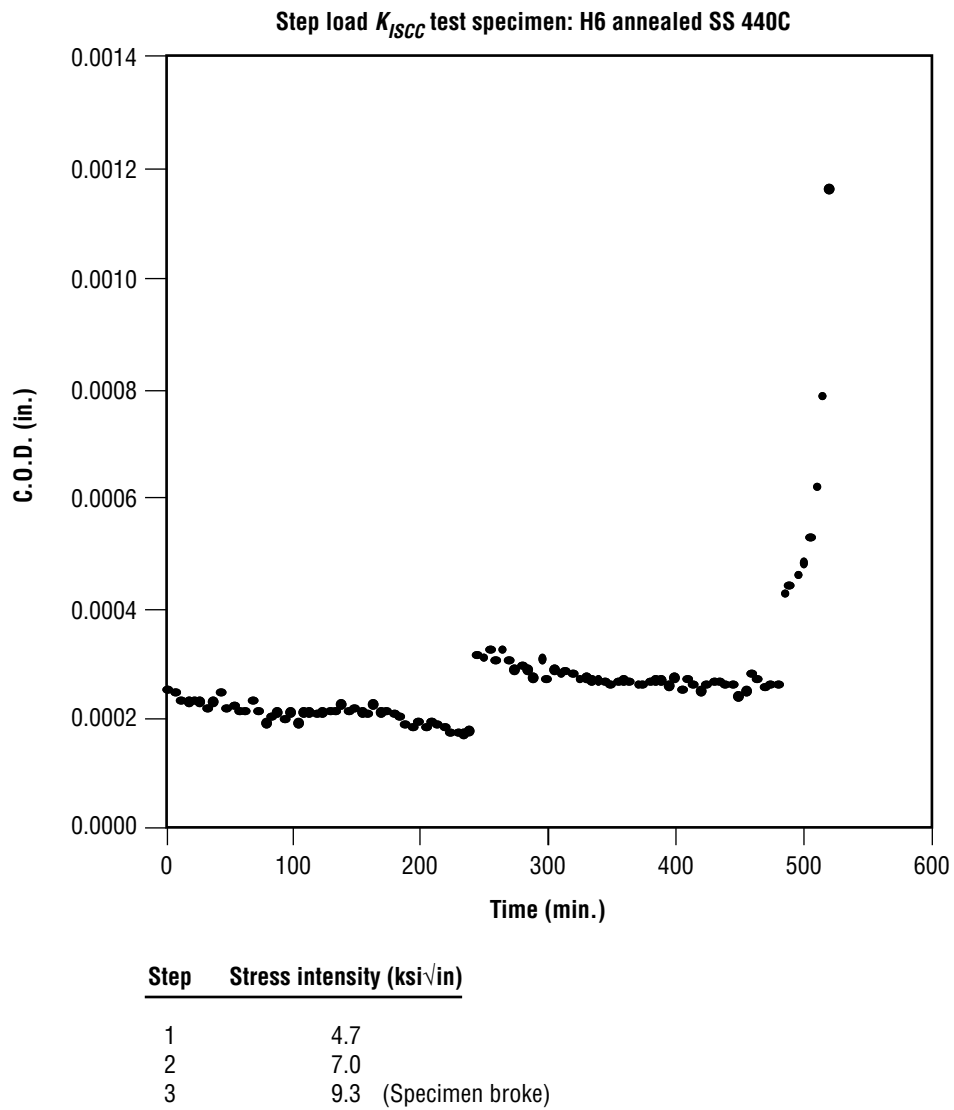
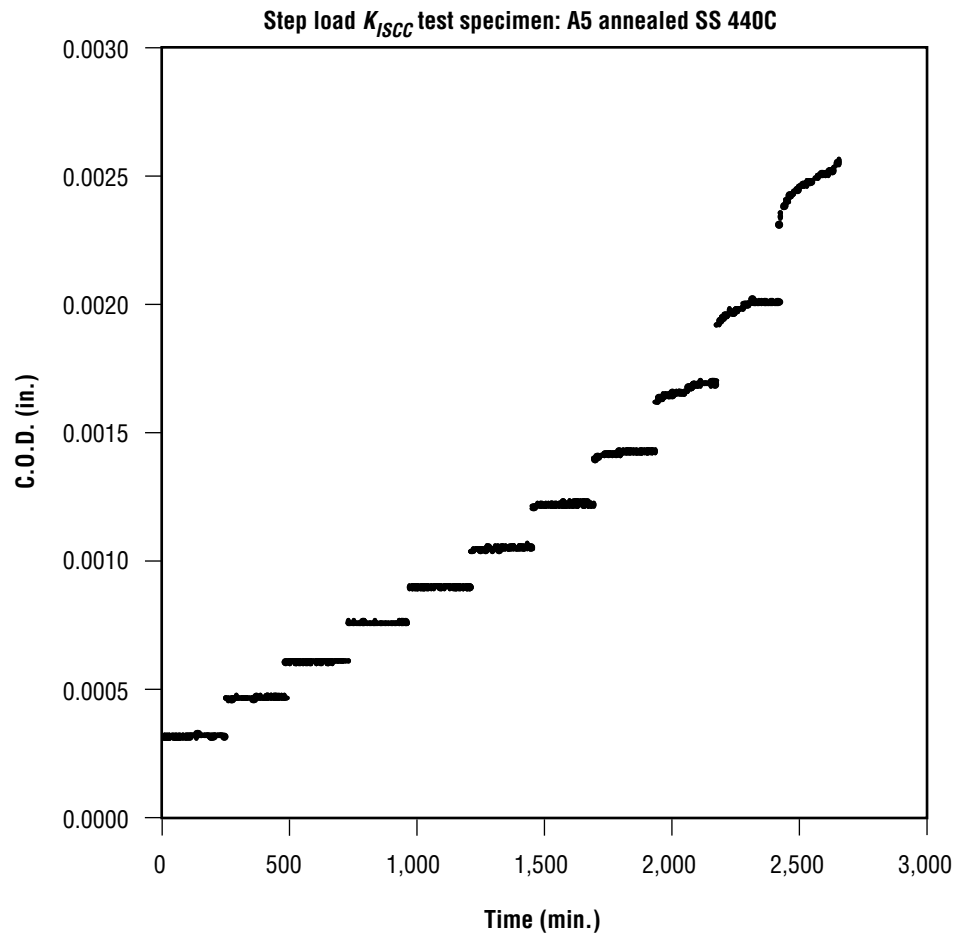
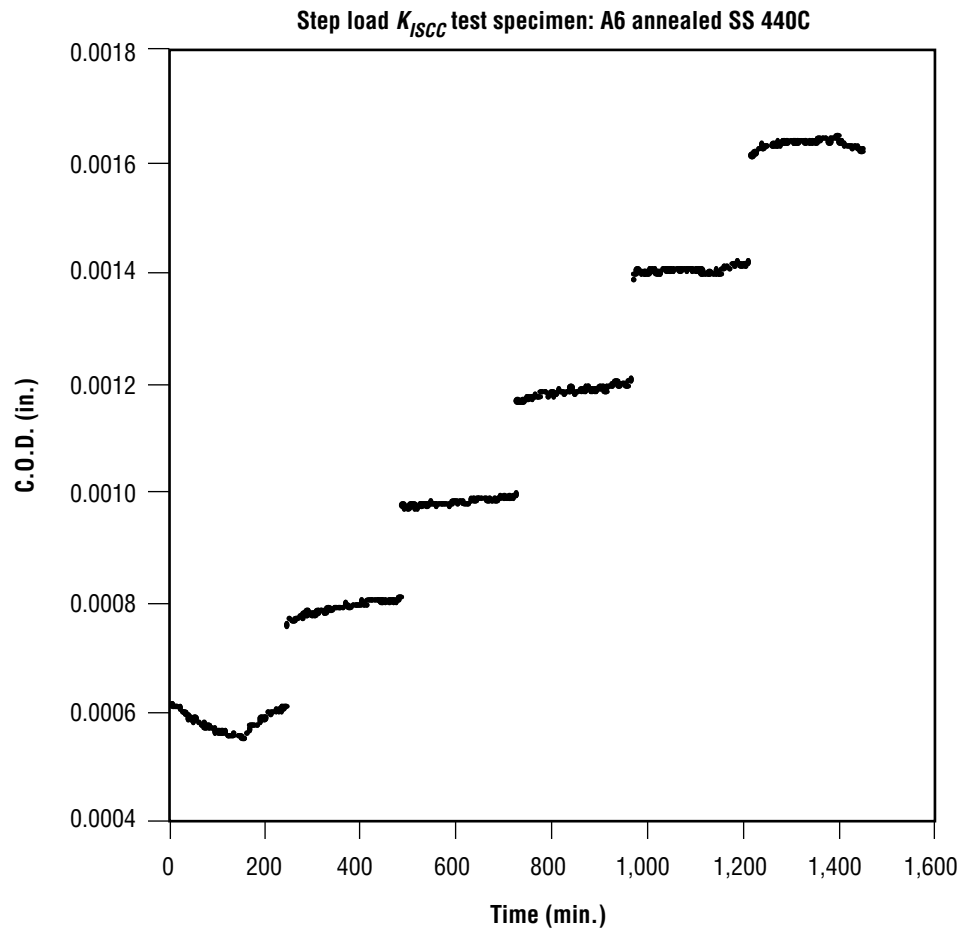


Figure 8. Incremental step load response of specimen H6 in 3.5-percent NaCl.



Step	Stress intensity (ksi√in)	Step	Stress intensity (ksi√in)
1	5.5	7	16.6
2	7.4	8	18.4 (Cracking starts)
3	9.2	9	20.2
4	11.0	10	22.1
5	12.9	11	23.9
6	14.7	12	25.8 (Specimen broke)

Figure 9. Incremental step load response of specimen A5 in 3.5-percent NaCl.



Step	Stress intensity (ksi√in)
1	10.3
2	12.4
3	14.5
4	16.6
5	18.6
6	20.7
7	22.8 (Specimen broke)

Figure 10. Incremental loading response of specimen A6 in 3.5-percent NaCl.

#### 4.4.3 The Constant Load Rate Technique

Data generated using the constant load rate method are shown in figures 11 and 12 for the hardened and annealed materials, respectively. Offset values were determined from deviations from the linear portion as described in the procedures section. The load and stress intensity corresponding to the 1-, 2-, and 4-percent offsets are given in figures 11 and 12. The upper bound and lower bound values noted represent the percent of the full range tested from which the linear portion of the plot was derived. The correlation coefficient ( $R^2$ ) for the linear portion is also given.

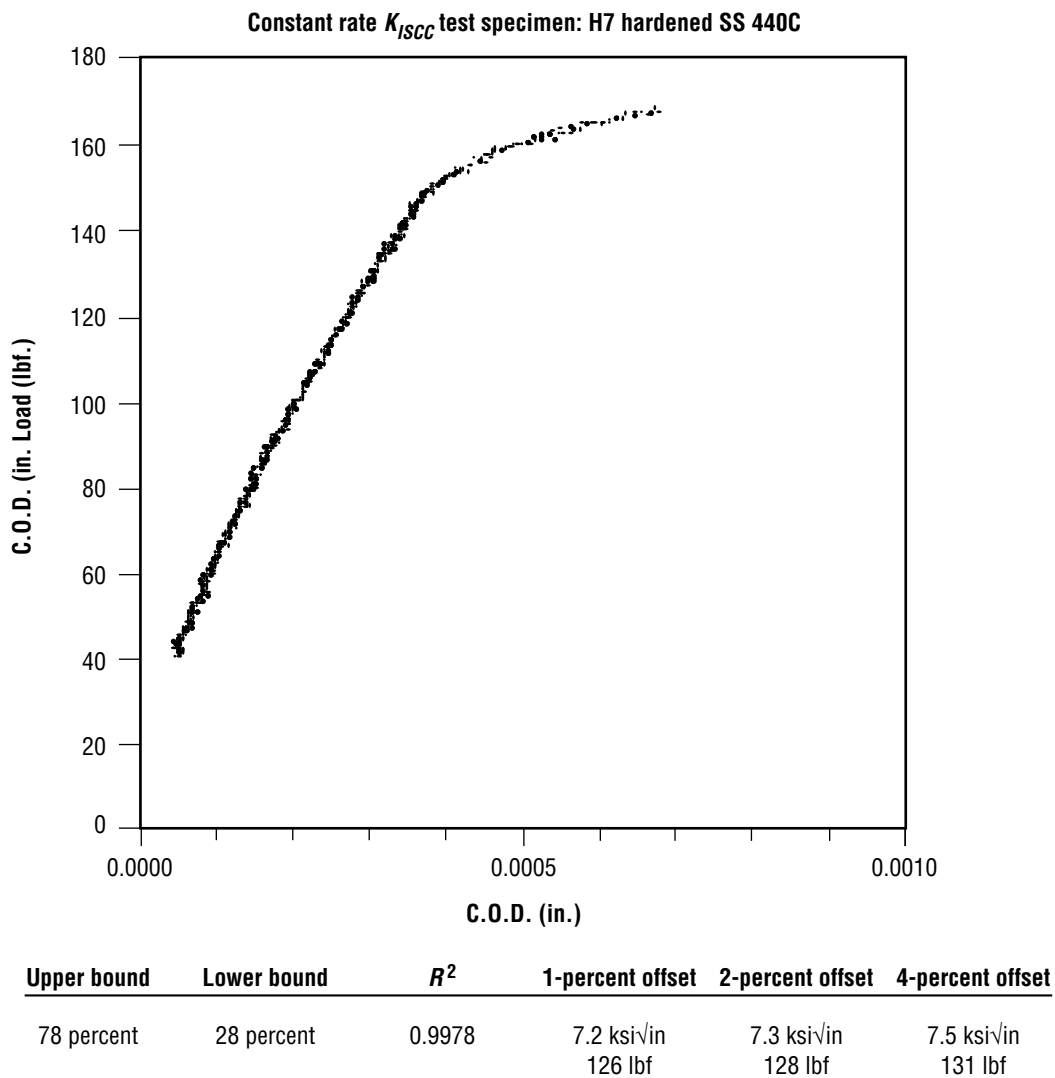


Figure 11. Constant loading rate response of specimen H7 in 3.5-percent NaCl.

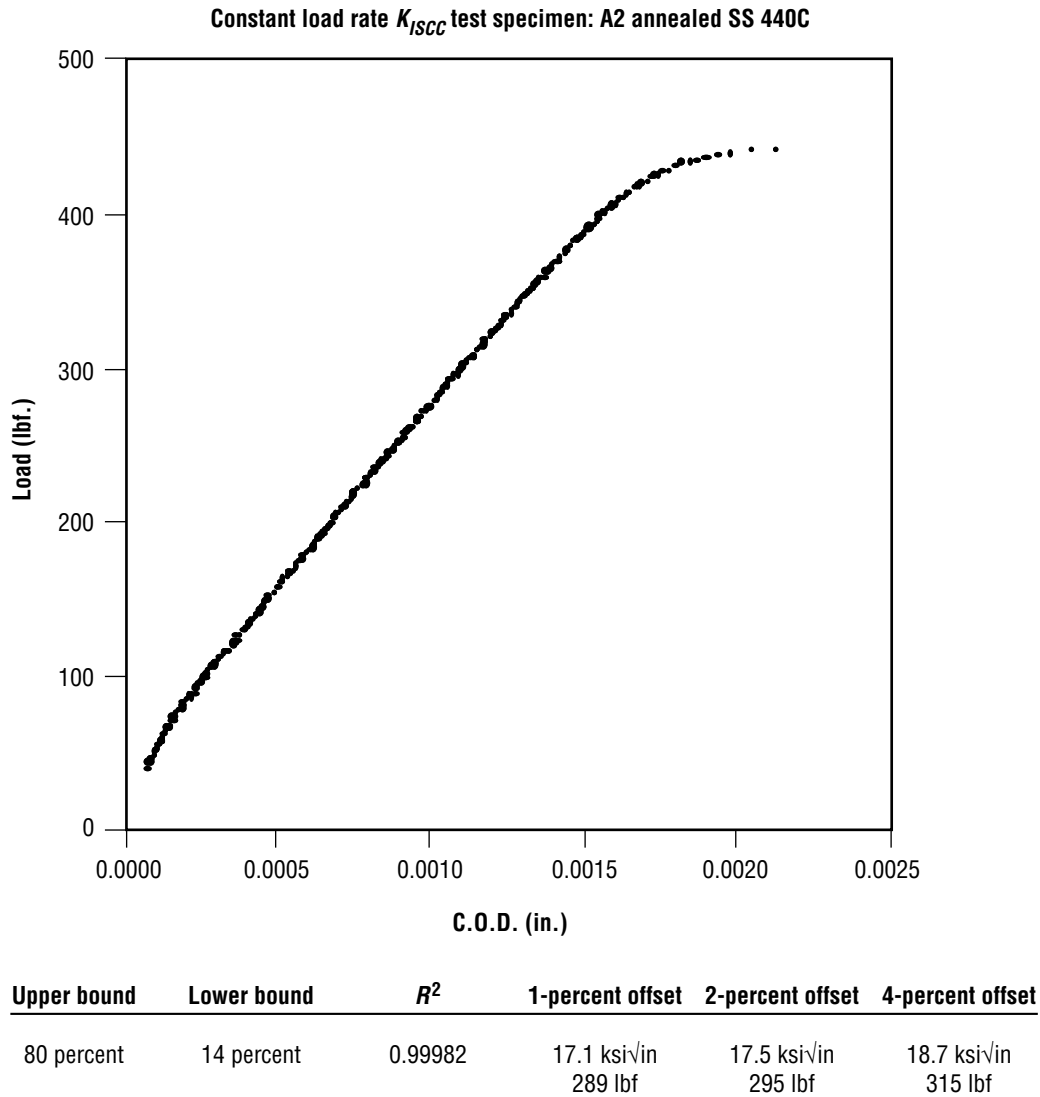


Figure 12. Constant loading rate response of specimen A2 in 3.5-percent NaCl.

A summary of the data for both techniques is given in table 2. Note that the results are reported as  $K_{QSCC}$  since it is uncertain whether these values represent true stress corrosion thresholds ( $K_{ISCC}$ ). Since the incremental step load method yields less conservative data, it is unlikely that these values represent true thresholds.

#### 4.4.4 Scanning Electron Microscopy

Photographs from failed specimens are shown in figures 13-19.

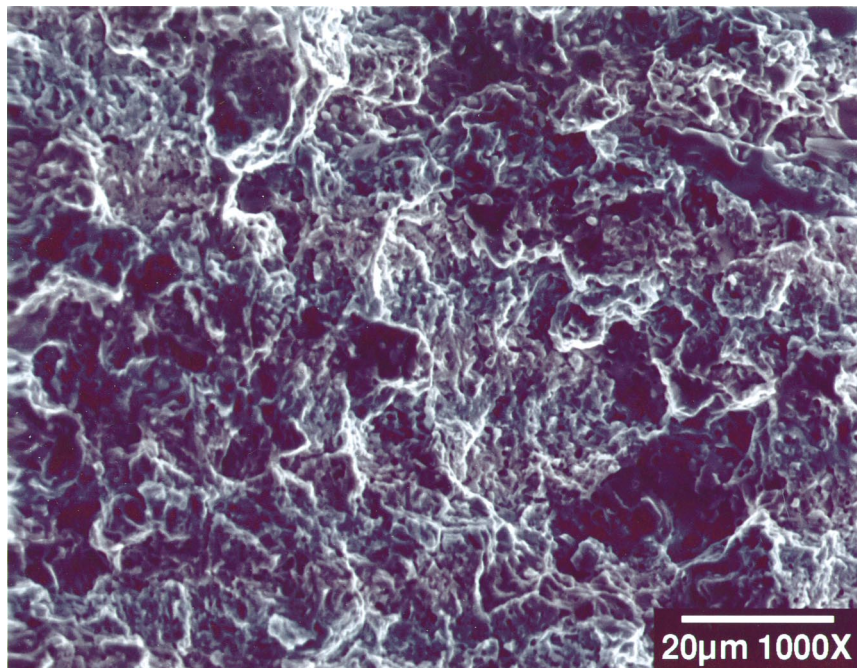
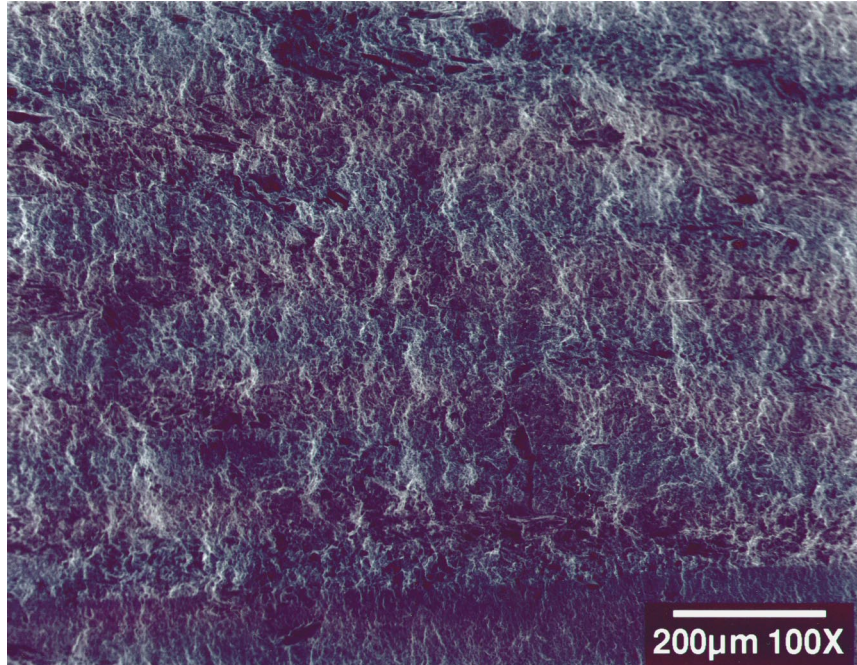


Figure 13. SEM photograph of specimen H1 (50-percent lactic/50-percent phosphoric acid etch).



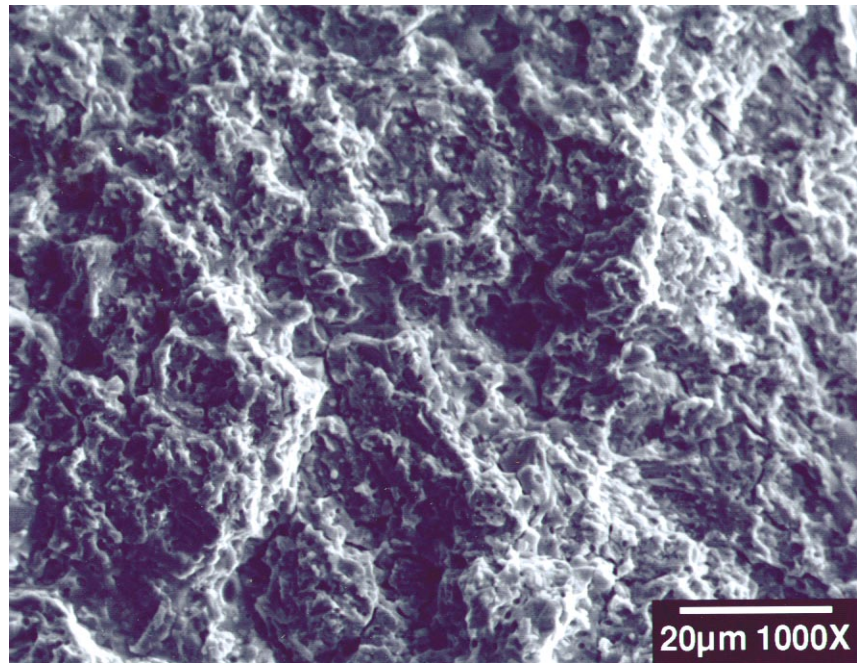
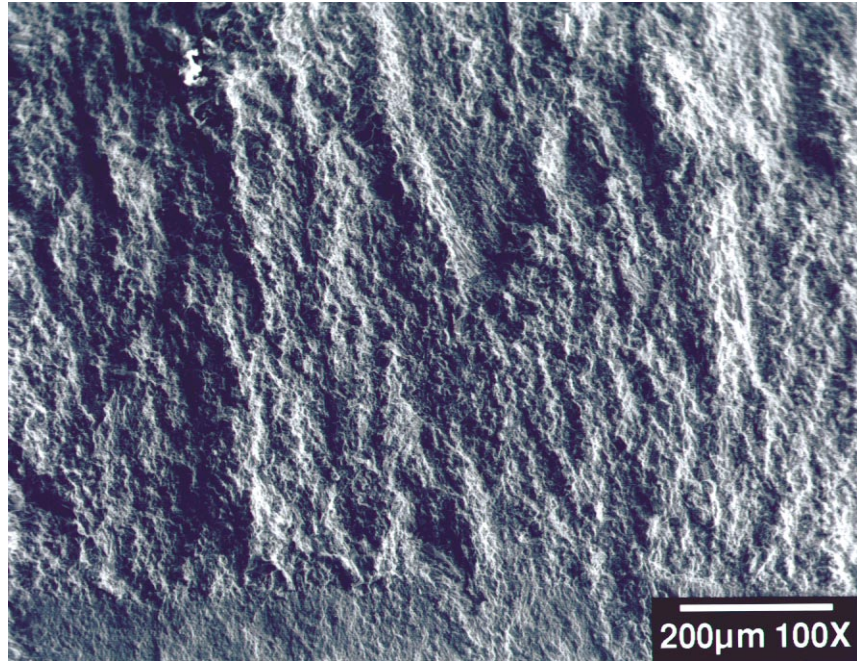


Figure 14. SEM photograph of specimen H2 (50-percent lactic/50-percent phosphoric acid etch).



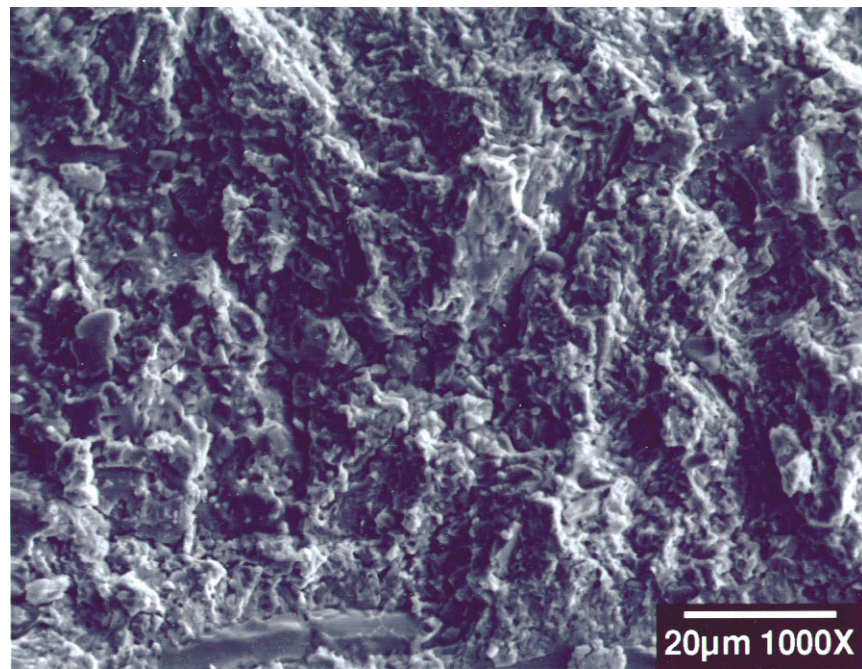
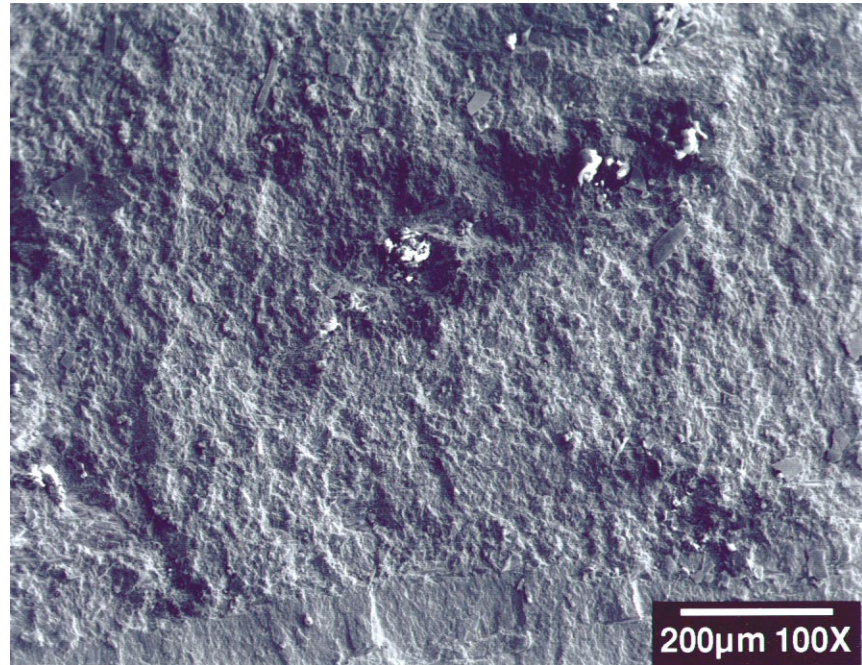


Figure 15. SEM Photograph of specimen H6 (50-percent lactic/50-percent phosphoric acid etch).



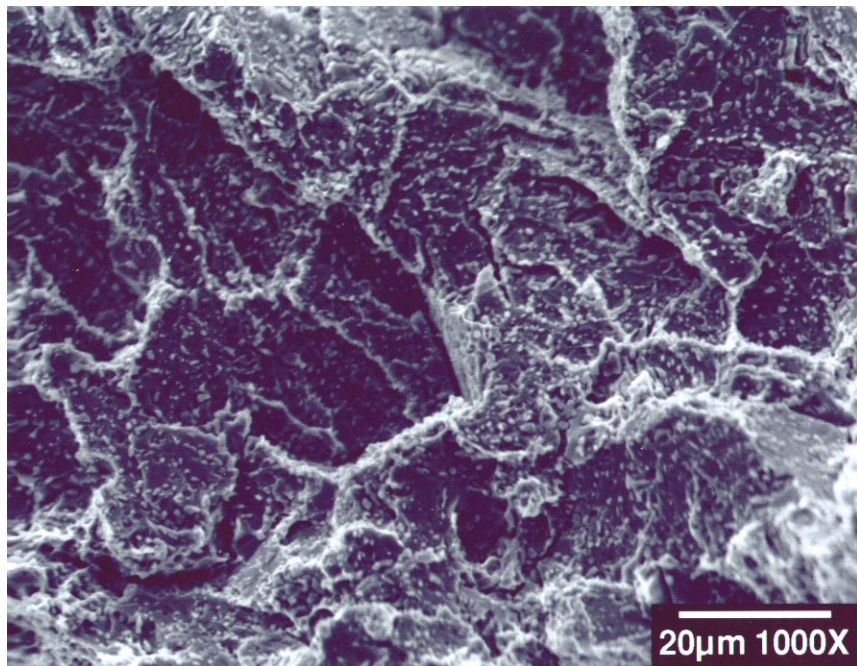
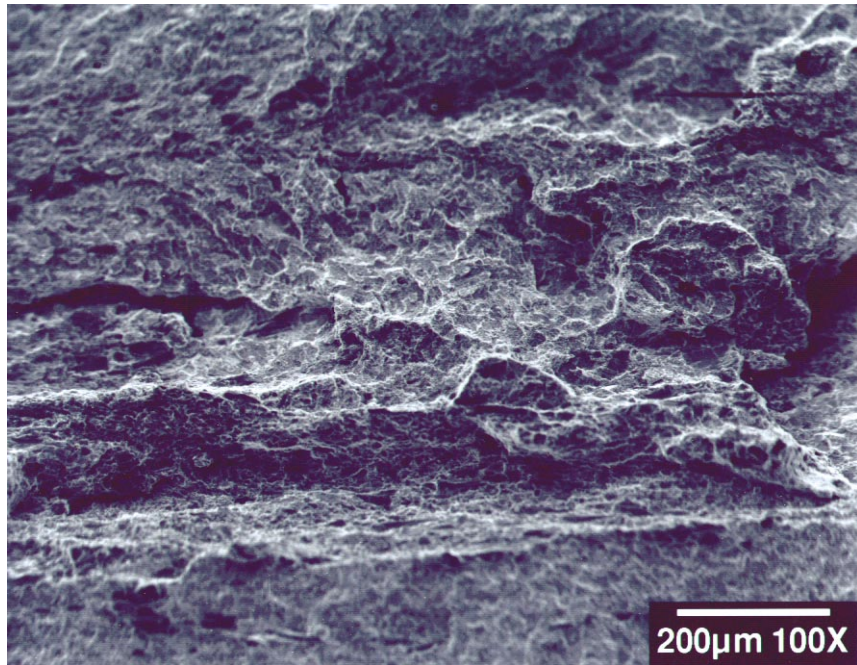


Figure 16. SEM photograph of specimen A5 (50-percent lactic/50-percent phosphoric acid etch).



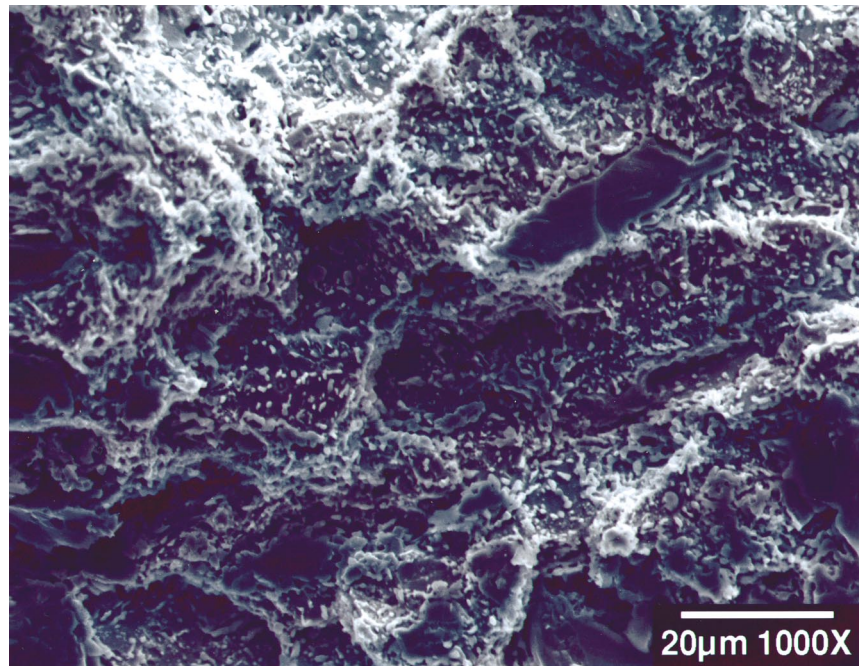
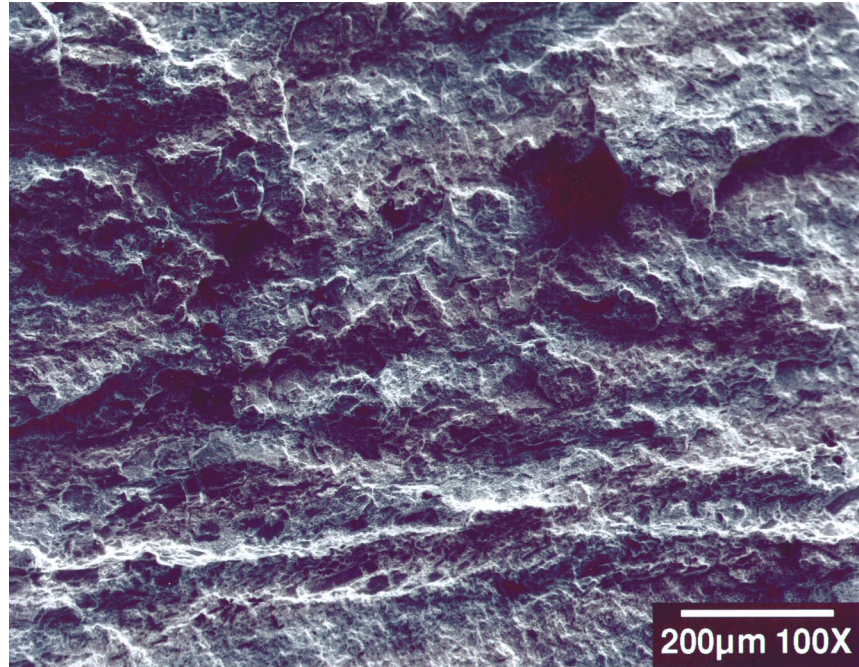


Figure 17. SEM photograph of specimen A6(50-percent lactic/50-percent phosphoric acid etch).



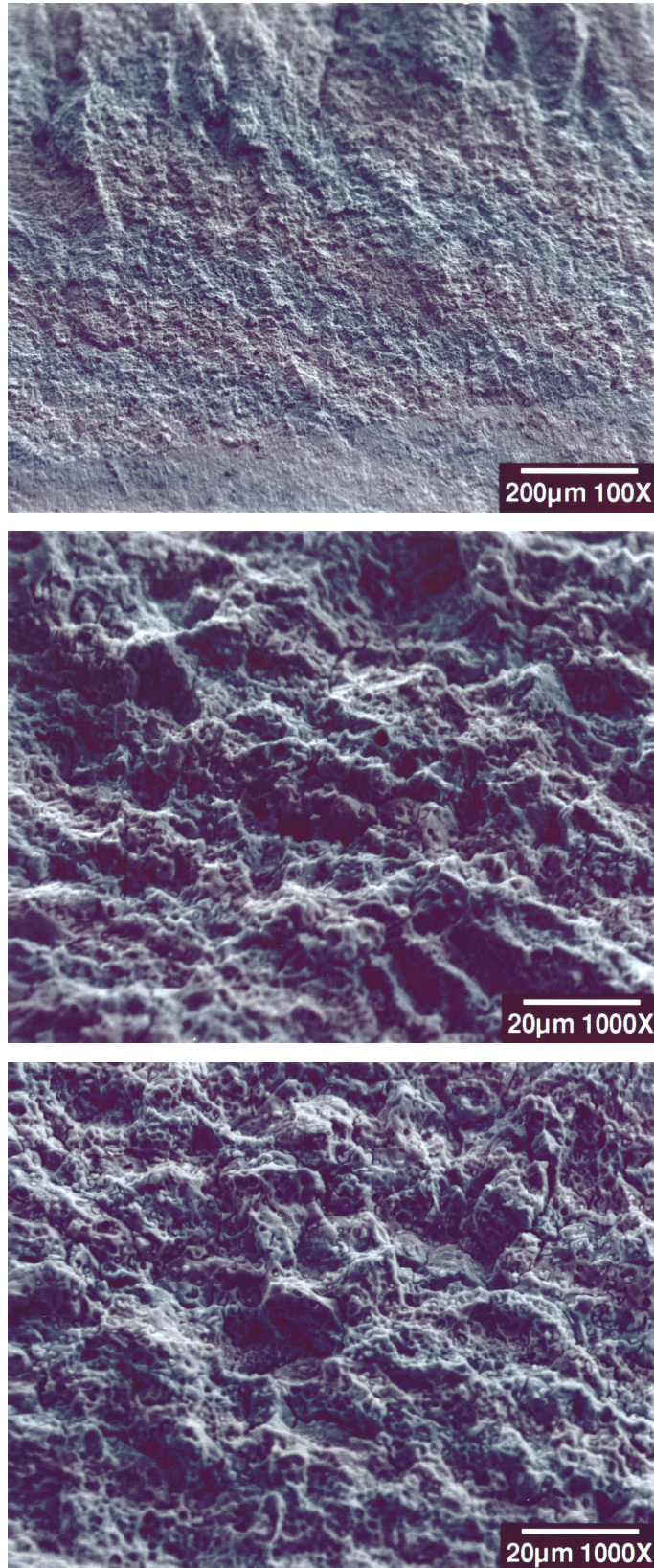


Figure 18. SEM photograph of specimen H7 (50-percent lactic/50-percent phosphoric acid etch).



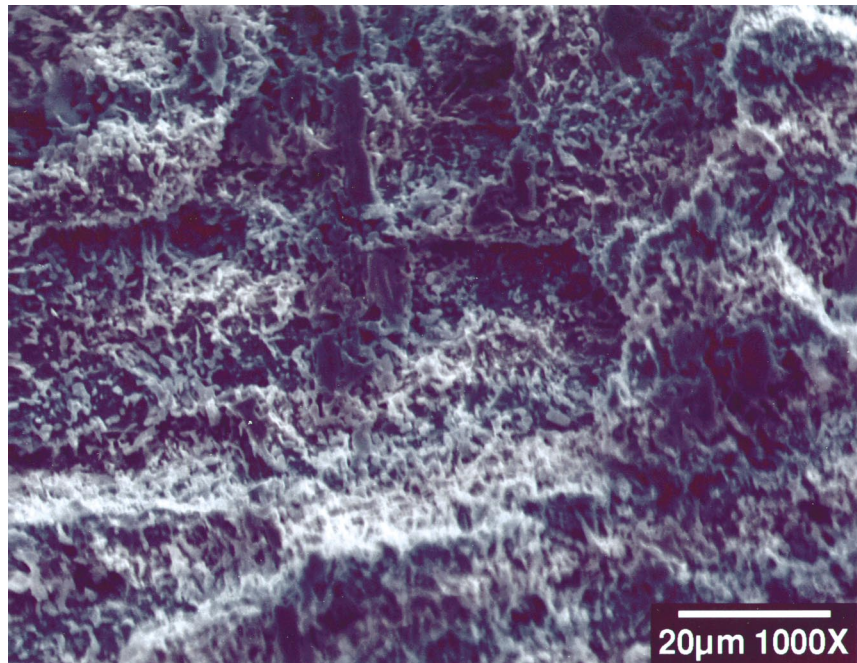
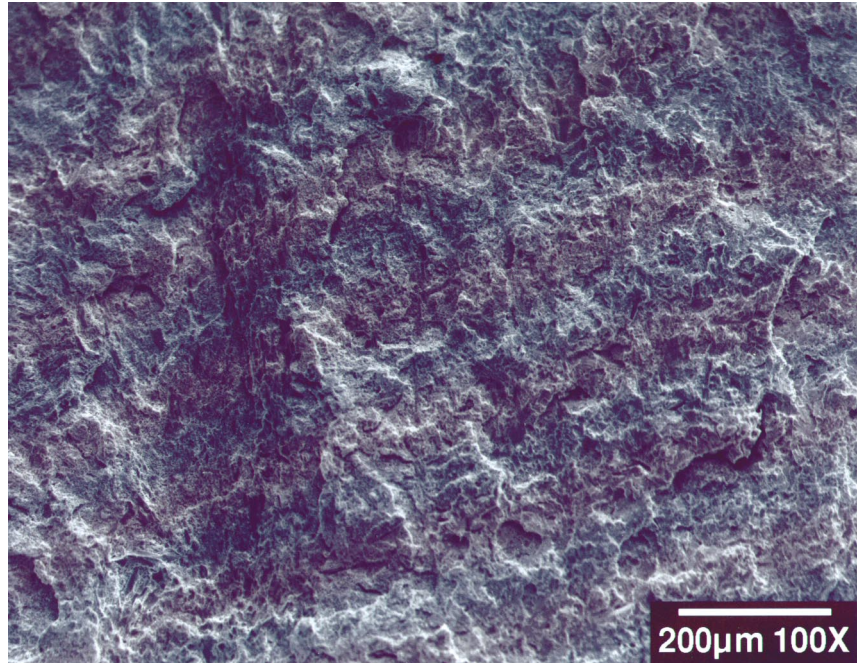


Figure 19. SEM photograph of specimen A2 (50-percent lactic/50-percent phosphoric acid etch).

## 5.0 DISCUSSION

The general corrosion testing indicates very little difference between annealed and hardened 440C. It is possible to conclude from the photographs that the hardened material exhibits slightly better resistance. However, the majority of the difference noted on the bare panels could be attributed to the annealed panel lying in the horizontal position for up to 3 days during exposure. Grease-plated samples show little difference in the frequency and size of corrosion pits, although the frequency appears to be slightly higher on the hardened sample and the size of corrosion pits slightly larger on the annealed sample.

The electrochemical corrosion testing, on the other hand, provides conclusive evidence that the annealed material is more susceptible to corrosion than the hardened material by an order of magnitude. Based on these results, it is unlikely that the annealed panel in 100-percent humidity lying flat had much to do with the greater level of attack on that panel.

The constant strain SCC testing shows that the annealed material has superior SCC resistance compared to hardened 440C. However, had the test been terminated at 60 days, per the test plan, no conclusion could have been drawn since only one specimen failed. Since this specimen was hardened 440C that failed after only 1 day, it would be difficult to conclude that the failure was caused by SCC and not simply due to growth of an existing defect after loading.

The precracked specimens, on the other hand, yield conclusive results. Data for the hardened material using the incremental step load method consistently show crack growth in the range of 7-10 ksi-in.<sup>1/2</sup>. Analogous data for the annealed material show that crack growth does not begin until a stress intensity of 18-23 ksi-in.<sup>1/2</sup> is reached. This indicates that the annealed material is significantly more resistant to SCC than the hardened material.

Results from the constant load rate test are more conservative, but agree well with the incremental step load method. The 1-, 2-, and 4-percent offset values for the annealed material are significantly higher than those of the hardened material. Whether these values represent SCC thresholds can only be determined by the more conventional WOL and cantilever beam type testing.

Using average values of 19.6 and 8.4 ksi-in.<sup>1/2</sup> for the annealed and hardened materials respectively, along with  $K_{IC}$  values for both materials, it is possible to propose criteria for SCC resistance based on the ratio of the stress intensity required for crack propagation in a corrosive environment to that required in air. These values are given in table 3 and demonstrate the superior resistance of the annealed 440C material.

Since it is quite possible that the  $K_{QSCC}$  data generated do not represent true threshold values, it is obvious from figures 6-12 that these techniques will require some refinement. For example, a more sensitive COD gauge should help reduce some of the drift noted in the step load testing. It may also improve the linearity of the load versus COD plot in the constant load rate testing. The goal of this work is to present the

feasibility of these techniques and propose further study, leading to written procedures that may be tested in other laboratories. Ultimately, the desire is to establish more objective criteria for rating the stress corrosion cracking susceptibility of structural materials and provide data that may be used by designers in practical applications.

## **6.0 CONCLUSIONS**

Hardened 440C stainless steel exhibits general corrosion resistance superior to annealed 440C.

Annealed 440C stainless steel exhibits superior SCC resistance to hardened 440C.

## **7.0 RECOMMENDATIONS**

Pursue the development of the techniques described in this report and develop written procedures. Establish interlaboratory verification of these procedures. If feasible, develop an ASTM standard for SCC testing using these methods. Rewrite MSFC-SPEC-522, incorporating the new methods and the data generated from them, along with objective criteria for the rating of material susceptibility to SCC. Incorporate environment specificity into the 522 document, so that ratings for materials tested in aggressive environments are not used to determine the suitability of a material for use in a more benign environment.

## APPENDIX

Table 1. Time to Failure of Constant Strain Stress Corrosion Specimens.

Condition	Hardened		Annealed	
Stress Level (ksi)	30	43.6	30	43.6
Days to Failure	166,263,324	1, 130, 152	n.f.	n.f.

n.f. – No failure

Table 2. Summary of Results from Incremental Step Load and Constant Load Rate Testing.

Specimen	Condition	Test Method	$K_{QSCC}$ (ksi-in <sup>1/2</sup> )
H1	Hardened	Step load	7.0
H2	Hardened	Step load	10.0
H6	Hardened	Step load	9.3
A5	Annealed	Step load	18.4
A6	Annealed	Step load	22.8
H7	Hardened	Constant strain rate	7.3*
A2	Annealed	Constant strain rate	17.5*

\* Represents the 2-percent offset data point

Table 3. Proposed Rating System for SCC Susceptibility.

Material	$K_{IC}$ in air (ksi-in <sup>1/2</sup> )	$K_{QSCC}$ (ksi-in <sup>1/2</sup> )	$K_{QSCC}/K_{IC}$
Annealed 440C	25.2	19.6	0.78
Hardened 440C	19.4	8.4	0.43

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